

OPTIMIZATION OF THE PARAMETERS INFLUENCING THE INHIBITION EFFICIENCY OF 6063 AL ALLOY IN 0.5 M NaOH USING RESPONSE SURFACE METHODOLOGY

P. R. PRABHU¹, DEEPA PRABHU² & PADMALATHA. RAO³

¹Associate Professor, Department of Mechanical and Manufacturing Engineering, Manipal Institute of Technology,
Manipal Academy of Higher Education, Manipal, Karnataka, India

²Assistant Professor Senior Scale, Department of Chemistry, International Center for Applied Sciences,
Manipal Academy of Higher Education, Manipal, Karnataka, India

³Professor, Department of Chemistry, Manipal Institute of Technology, Manipal Academy of Higher Education, Manipal,
Karnataka, India

ABSTRACT

In this study, the influence of two process parameters (concentration of inhibitor and temperature) on the corrosion current density and inhibition efficiency of 6063 Al alloy in 0.5 M NaOH environments using Coriandrum Sativum L. Extract (CSE) were examined by response surface methodology (RSM). The parameters considered for the study were found in 5 levels: temperature (X_1) at 30, 35, 40, 45 and 50°C and concentration of inhibitor (X_2) at 0.1, 0.2, 0.3, 0.4 and 0.5 g/l was taken to correlate the parameters with the inhibition efficiency as an output parameter. A regression model was established and authenticated before the parameters were optimized for the highest inhibition efficiency. The results revealed that the concentration of inhibitor has the significant effect on the inhibition efficiency followed by temperature, and the data predicted by regression analysis had a good agreement with the data obtained from the experiments with the values of $R^2 = 0.9921$ and $\text{Adj-}R^2 = 0.99$ for inhibition efficiency. The optimum settings for the studied parameters were found to be at temperature (30°C) and concentration of inhibitor (0.5 g/l) to achieve maximum inhibition efficiency of 74.11%. The study has also shown that the data obtained from response surface design is an effective technique for forecasting the optimum parameter setting required to maximize inhibition efficiency of 6063 Al alloy in 0.5 NaOH solution by incorporating all parameters under consideration.

KEYWORDS: Coriandrum Sativum L., ANOVA, Regression Analysis, Response Surface Methodology, NaOH Environment & Optimization

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1. INTRODUCTION

In most of the industries, for preventing the acid corrosion, organic inhibitors are extensively used for some of the processes like descaling, pickling, cleaning and oil well acidizing[1-3]. The efficiency of organic inhibitor is carefully related to adsorptive association with the outside of the metal, through electron lavish practical gatherings. The sulfur-containing mixes are generally recognized to have higher effectiveness among this sort of inhibitors[4-6]. Ecological concerns are pushing forward the examinations, directing to create various inhibitors, so ecofriendly inhibitors have been as often as possible considered [7-10]. For the said reason, the utility ought to likewise be improved and enhanced to utilize less synthetic substances in down to earth applications. In any form, the natural conditions display irregularity in a precise range. In this way, all

significant parameters (pH, the concentration of inhibitor, temperature) must be kept at an optimum level for the success of the inhibitor application.

The conventional way of conducting the experiments is becoming out dated, because it includes changing one parameter at a time by keeping other parameters fixed. The other option, which consists of carrying out investigations with all possible combinations of variables, is often impracticable due to the large number of experimental runs it requires. RSM is a widely used technique for designing the experiments, modeling the process parameters, analyzing the effects of each parameter on the response, and determining the optimum parameter setting for the appropriate responses in various chemical processes [11-15]. It is desirable for conducting multi-factor experiments, in that it specifies the most common interactions between several factors for the determination of the most promising or adverse circumstances of the processes [16-17]. RSM is an instrumental and robust technique for examining the effect of several process parameters influencing the output parameter by changing them instantaneously and conducting only a few numbers of experiments [11]. The main benefit of chronicled data response surface design is that, it offers the user a chance to characterize the plan focuses on utilizing all or a portion of the available data [16]. Rather than different kinds of methods in RSM, there does not impede the number of parameters that can be provided in the design. The parameter settings and the existing data of any response can be directly brought to the fresh layout [11].

Hawraa Khaleel et al. used ANOVA to study the effect of temperature and concentration of inhibitor on the rate of corrosion of carbon steel in 1M HCl solution by computing the optimum parameters to minimize the corrosion rate from the developed mathematical model equations[18]. M.Sc. Maher T et al. used full factorial experiment to examine the influence of 3 process variables (the time of immersion, the concentration of inhibitor, and temperature) on the rate of corrosion of mild steel by computing the optimum parameter setting for the inhabitation efficiency using the extract of Ziziphus plant leaves in 3.5 NaCl solution[19]. Omoruwou F et al. studied the response surface modeling and optimization of inhibition efficiency of water hyacinth on mild steel in the acidic medium using central composite design (CCD)RSM to evaluate the influence of process parameters on corrosion rate and optimizing these parameters[20]. Femiana Gapsari et al. investigated the effects of environmental factors on the rate of corrosion of AISI 304 austenitic steel through potentiodynamic polarization, using CCD and RSM[21]. Sunday O. Ajeigbe et al. were investigated the effect of efficiency of vital oils of Alpinia galangal on mild steel in HCl solution using the weight loss method. The results of the concentration of inhibitor, temperature, and immersion time were optimized for maximum inhibition efficiency using RSM and CCD[22]. Khalid Hamid Rashid et al. investigated the effect of concentration of inhibitor, temperature, immersion time on medium carbon steel in salty water containing nitrate and chloride using sodium molybdate as a corrosion inhibitor. The primary and interaction effects of these factors were optimized for obtaining a minimum corrosion rate using CCD and RSM[23]. Deniz Bingöl et al. studied the influence of concentration sodium dodecyl benzene sulfonate solution and temperature in HCl solution on the corrosion rate of aluminum using the RSM and CCD[14].

Going by literature, the majority of the studies on corrosion used a weight-loss method to determine the corrosion rate and optimizing the parameters using response surface methodology. Very few studies employed the design of experiments for enhancing the inhibition efficiency by using electrochemical measurement techniques; hence, the need for this study. The objective of this work is to estimate the optimum parameter settings of NaOH environment in contact with 6063 Al alloy needed to minimize corrosion rate and maximize inhibition efficiency using central composite design with response surface methodology.

2 MATERIALS AND METHODS

2.1 Preparation of Inhibitor

Seeds of *Coriandrum sativum* L. were dried in an oven at 40 °C for 2 h. Then the dried seeds were powdered and refluxed in water for 3 h and filtered. Filtrates were warmed gradually on a water bath to evacuate water substance. After drying, the powder was then finely ground and protected in a desiccator.

2.2 Preparation of Metal Specimen

The commercially obtained sample of aluminum 6063 alloys was used for the study. The ingredients of 6063 Al alloy are shown in Table 1. The circular test coupon was fixed with acrylic gum material so that the zone presented to the medium was 1.0 cm². The specimen was cleaned with various then polished with emery papers of different grades and further washed with disc polishing machine utilizing alumina paste till we get a mirror-like surface finish on the specimen. It was then dried and transferred to a desiccator to maintain a strategic distance from dampness before being utilized for studies on corrosion.

Table 1: Composition of the 6063 Al Alloy

Element	Composition (weight %)
Si	0.412
Mg	0.492
Fe	0.118
Cu	0.057
Al	98.92

2.3 Corrosion Medium

A stock solution of NaOH was made ready by dissolving systematic evaluation (Merch) NaOH pellets in twofold refined water and standardized by volumetric method. Solutions of required qualities 0.5 M were set up by suitable thinning as and when required. Analyses were completed utilizing a standardized thermostat at temperatures 30°C, 35°C, 40°C, 45°C, and 50°C ($\pm 0.5^\circ\text{C}$).

2.4 Electrochemical Measurements

Tests were performed in exposed to 0.5 M NaOH solution with and without different concentrations of CSE at different temperatures. The 3-cathode framework was utilized for potentiodynamic polarization estimations. A counter electrode (Platinum electrode), a reference electrode (calomel electrode) and a working electrode (Al 6063 alloy). The working electrode was in the shape of a rod, cut from 6063 aluminum alloy acquired from a commercial supplier, and inserted in epoxy sap with the goal that the level surface territory of each working cathode was 1.0 cm².

2.5 Potentiodynamic Polarization (PDP) Measurements

Finely cleaned test coupons were presented to 0.5 M NaOH arrangement, with and without inhibitors of various concentrations at various temperatures (30°C to 50°C) and permitted to set up a consistent state open circuit potential for around 0.5 h. Test coupons were then spellbound by the use of potential float of -250mV cathodically and +250mV anodically concerning the OCP at a scan pace of 1.0 mVs⁻¹. Simultaneously, Tafel curves were then developed. The data of corrosion rate and inhibition efficiency is used for this RMS studies.

3. METHODOLOGY

3.1 Experimental Design

Typically, composite design is an examination technique that includes some unequivocal preliminaries on the basis of regression design points. Therefore, it can substantially diminish the number of trials. The process variables studied were temperature (X_1) and inhibitor concentration (X_2). These two parameters were considered at five levels. The settings and the levels of each of the parameters are as shown in Table 2. The Minitab 19 programming was utilized for the exploratory runs and displaying of trial information. Twenty-Five (25) tests were created with CCD (Central Composite Design), which is appeared in Table 3.

3.2 The Regression Model

As described above, the temperature and concentration of inhibitor have substantial effects on inhibition efficiency. In this examination, the full quadratic model is received to build up the connection between the inhibitor effectiveness and the process variables. The impacts of different process variables on the inhibition efficiency are precisely analyzed.

$$Y = b_0 + \sum_i^k b_i X_i + \sum_{i=1}^{k-1} \sum_{j=i+1}^k b_{ij} X_i X_j + \sum_{i=1}^k b_{ii} X_i^2 - - - - \quad (1)$$

For two factor inputs of X_1 and X_2 , the quadratic response is shown below;

$$Y = b_0 + b_1 X_1 + b_2 X_2 + b_{12} X_1 X_2 + b_{11} X_1^2 + b_{22} X_2^2 - \quad (2)$$

Y = response/output parameter

b_0 = constant-coefficient

b_1 and b_2 = linear effect coefficient

b_{11} and b_{22} = quadratic effect coefficients

b_{12} = interaction effect coefficients

The study aims to identify the optimum parameter setting to acquire the maximum inhibition efficiency. While as a general rule, it is tough to find out optimum processing variables. Henceforth, the RSM approach is used to examine the impacts of the different process parameters on the response. Then, the combination of the process variables that can accomplish an optimum anticipated value can be acquired.

Table 2: Levels of Experimental Parameters Selected for the CCD

Parameter	Symbol	Unit	Level of Parameters				
			Level 1	Level 2	Level 3	Level 4	Level 5
Temperature	X_1	°C	30	35	40	45	50
Inhibitor concentration	X_2	g/L	0.1	0.2	0.3	0.4	0.5

Table 3: Test Design Matrix and Levels Dependent on the CCD in Terms of Actual Variables

Run Order	Actual Level of Parameter	
	X_1	X_2
1	30	0.1
2	30	0.2
3	30	0.3
4	30	0.4

5	30	0.5
6	35	0.1
7	35	0.2
8	35	0.3
9	35	0.4
10	35	0.5
11	40	0.1
12	40	0.2
13	40	0.3
14	40	0.4
15	40	0.5
16	45	0.1
17	45	0.2
18	45	0.3
19	45	0.4
20	45	0.5
21	50	0.1
22	50	0.2
23	50	0.3
24	50	0.4
25	50	0.5

4. RESULTS & DISCUSSIONS

The result of the response surface methodology plan of investigation is presented in Table 4. The corrosion current density is achieved from testing the polarization potentiodynamic. The Tafel plots of potentiodynamic polarization testing to get the corrosion rate and inhibition efficiency at 30 °C for 6063 aluminum alloy are presented in Figure 1.

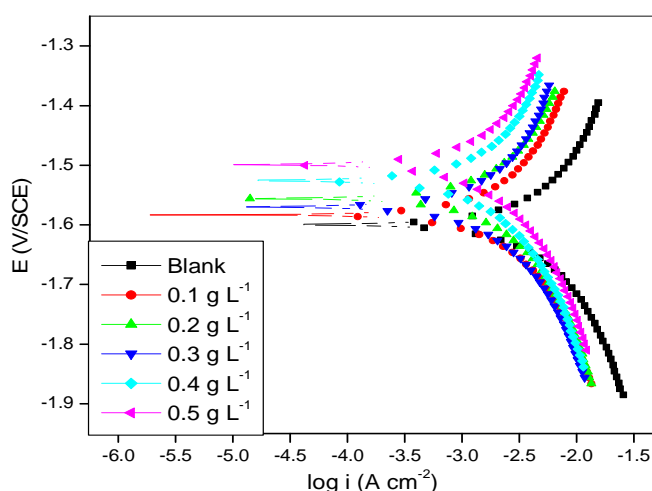


Figure 1: Tafel Plots for 6063 Aluminum Alloy with CSE Inhibitor at 30 °C in 0.5 M NaOH Environment.

Screening tests demonstrated that the concentration of CSE inhibitor and temperature had a major impact on the corrosion current density (i_{corr}). This way, these two parameters were chosen for detailed experimentation. Methods studied the impacts of temperature (X_1) and concentration of CSE inhibitor (X_2) for a Central Composite Design (CCD). The investigations were performed haphazardly to keep away from orderly mistakes, and the outcomes were analyzed utilizing Minitab-19 programming. All the actual and coded values of the process parameters appear in Table 1. The experimental design and the levels dependent on the CCD of individual parameters are given in Table 2 as actual values alongside the

output parameter (Corrosion current thickness, i_{corr}). The efficiency of the inhibitor acquired from polarization results, utilizing the following equation:

$$IE (\%) = \frac{i_{corr}^b - i_{corr}}{i_{corr}^b} \times 100 - \quad (3)$$

Where, i_{corr}^b and i_{corr} are the corrosion current densities in the blank and inhibited solutions, respectively.

The inhibitor concentration and temperature can alter the interaction between the surface condition of the working cathode and the acidic medium. Corrosion current density values obtained from polarization curves are exhibited in Table 2. It is seen that the corrosion current density decreased with an increase in the concentration of the CSE inhibitor.

From table 3, it has been witnessed that the largest efficiency is obtained in the experiment with an extract of 0.5 g/L CSE and with a working temperature of 30 °C. To find out the right combination for achieving the lowest corrosion current density with the highest efficiency of the two parameters of this study, RSM is used for the optimization analysis. At that point, the regression equation between the process variables and the inhibition efficiency was set up. The regression model, as far as coded factors for inhibition efficiency, could be stated utilizing the equation shown below:

$$\begin{aligned} \text{Inhibition Efficiency } (\%) = & 50.7 - 0.483 \text{ Temperature} + 67.3 \text{ Concentration of Inhibitor} - 0.00667 \\ & \text{Temperature*Temperature} + 39.3 \text{ Concentration of Inhibitor*Concentration of Inhibitor} + 0.065 \\ & \text{Temperature*Concentration of Inhibitor} \end{aligned} \quad (4)$$

Table 4: Actual and Predicted values of corrosion current Density and inhibition Efficiency values obtained from the Potentiodynamic Polarization curves and the Central composite Design

Run Order	Actual Level of Parameter		Corrosion Current Density (i_{corr})	Inhibition Efficiency (%)		Residual	% Error
	X ₁	X ₂		Experimental	Predicted		
1	30	0.1	2.02	37.91	37.53	0.38	1.00
2	30	0.2	1.75	46.33	45.63	0.70	1.51
3	30	0.3	1.54	52.81	54.52	-1.71	-3.24
4	30	0.4	1.14	65.01	64.20	0.81	1.25
5	30	0.5	0.84	74.11	74.66	-0.55	-0.74
6	35	0.1	2.52	32.74	32.97	-0.23	-0.70
7	35	0.2	2.19	41.36	41.11	0.25	0.60
8	35	0.3	1.95	47.87	50.03	-2.16	-4.51
9	35	0.4	1.43	61.7	59.74	1.96	3.18
10	35	0.5	1.09	70.93	70.24	0.69	0.97
11	40	0.1	3.04	28.33	28.09	0.24	0.85
12	40	0.2	2.69	36.61	36.26	0.35	0.96
13	40	0.3	2.38	43.89	45.22	-1.33	-3.03
14	40	0.4	1.83	56.86	54.96	1.90	3.34
15	40	0.5	1.49	64.92	65.48	-0.56	-0.86
16	45	0.1	3.5	23.11	22.87	0.24	1.04
17	45	0.2	3.11	31.67	31.08	0.59	1.86
18	45	0.3	2.8	38.52	40.06	-1.54	-4.00
19	45	0.4	2.21	51.47	49.84	1.63	3.17
20	45	0.5	1.9	58.22	60.40	-2.18	-3.74
21	50	0.1	4.55	17.19	17.32	-0.13	-0.76
22	50	0.2	4.09	25.67	25.56	0.11	0.43
23	50	0.3	3.7	32.65	34.58	-1.93	-5.91
24	50	0.4	2.88	47.68	44.38	3.30	6.92
25	50	0.5	2.52	54.21	54.98	-0.77	-1.42

The linear mathematical model is the main stage in the activity of the response surface methodology approach. Based on the experimental results, it was discovered that the outcome didn't match the fitted linear model. Therefore, the full quadratic model testing was taken into account for further investigation. Because of the CCD structure, ANOVA for the full quadratic response was found, as appeared in table 5.

To locate a decent comprehension of ANOVA, the usual impact of parameters for each level and the sum of squares (SS) and relating computations need further depiction. The level of significance utilized in this investigation is 5%. Given Table 3, the p-value is 0.000 in most of the cases. The quadratic model is noteworthy in light of the fact that their p-value is less than 0.05 indicates that the full quadratic model is the correct model for this case. The p-value of < 0.0001 for the model demonstrates that the model is appropriate. As can be seen in Table 3, four of the five p-values of the model are under 0.05, indicates that 80% of the model process parameters are having a significant effect on the inhibition efficiency.

Figure 2 represents a Pareto chart for 6063 aluminum alloy in 0.5M NaOH solution suggests that the dominant process factor is the concentration of inhibitor and the temperature. The interaction parameters and the quadratic parameters have a very less significant influence.

Table 5: Analysis of Variance for Inhibition Efficiency

Source	DF	Adj SS	Adj MS	F-Value	P-Value	% Contribution
Model	5	5621.75	1124.35	475.74	0.000	
Linear	2	5608.90	2804.45	1186.62	0.000	
Temperature	1	1241.51	1241.51	525.31	0.000	21.91
Concentration of Inhibitor	1	4367.39	4367.39	1847.93	0.000	77.07
Square	2	12.75	6.37	2.70	0.093	
Temperature*Temperature	1	1.95	1.95	0.82	0.376	0.03
The concentration of Inhibitor*Concentration of Inhibitor	1	10.80	10.80	4.57	0.046	0.19
2-Way Interaction	1	0.10	0.10	0.04	0.835	
Temperature*Concentration of Inhibitor	1	0.10	0.10	0.04	0.835	0.001
Error	19	44.90	2.36			0.79
Total	24	5666.66				

S	R-sq	R-sq(adj)	R-sq(pred)
1.53733	99.21%	99.00%	98.72%

DF: degrees of freedom, Seq SS: sequential sum of squares, Adj MS: adjusted mean of squares, P: probability

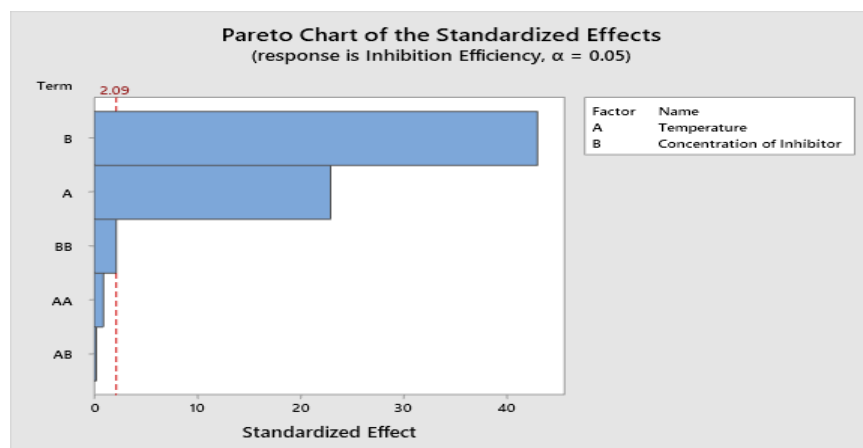


Figure 2: Pareto Chart for 6063 Aluminum Alloy.

4.1 Model Validation

Table 4 demonstrates the anticipated inhibition efficiency against the inhibition efficiency, which was obtained experimentally. The analysis gave R^2 of 0.9921 and adjusted- R^2 of 0.99, which are roughly near 1; in this way, showing a generally decent relationship between the anticipated and test estimations of the inhibition efficiency. Additionally, a maximum deviation of $\pm 3.32\%$ was obtained between the experimental and predicted inhibition efficiency, demonstrates there is a good fit between them.

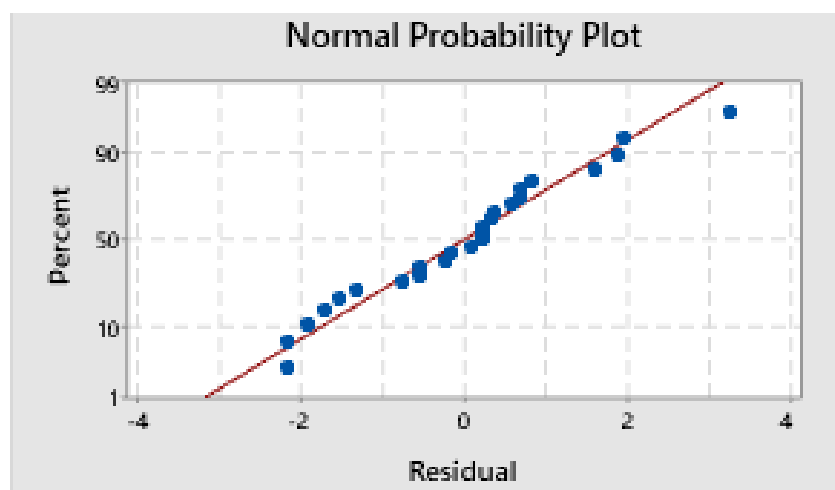


Figure 3: Normal Probability Plot for the Model.

Figure 3 demonstrates the graphical examination of the model, as did by the normal plot of the residuals. The residuals depict a typical dispersion because every one of the focuses pursues a straight line bend. Likewise, the residuals demonstrate that no further improvement should be possible to the model by making modifications to the response in light of the fact that the data focuses are dispersed and don't show a "S-molded" bend [13]. Accordingly, these figures and charts outline that the model in Equation (4) can be viewed as an ideal model for testing the 6063 aluminum alloy corrosion in NaOH condition. Henceforth, they will be utilized in finding the optimum process parameter settings in the NaOH environment.

The "Main effect" graphs are the additional output received from the regression analysis. Figure 4 demonstrates the main effect plots for the means of inhibition efficiency. A "Main impact plot" indicates how to control elements that influence the output parameter. A fundamental impact is speaking to when different degrees of a factor affect the response in an unexpected way. As per Figure 3, among the considered factor levels, the maximum inhibition efficiency was observed with 0.5 g/L concentration of CSE inhibitor and a temperature of 30 °C. Along these lines, it was anticipated that a blend under the previously mentioned conditions may have high inhibition efficiency and can be an appropriate definition for the considered inhibitor blend.

The interaction between factors occurs when the response change is different for two variables. Figure 5 shows the interaction plot for inhibition efficiency suggests that the interactions between the parameters are not very significant. From the ANOVA analysis, it is also seen that both square and two way interactions between the settings are not substantial because the p-value is higher than 0.05; demonstrating there is no inter-correlation between the inhibitor concentration and temperature.

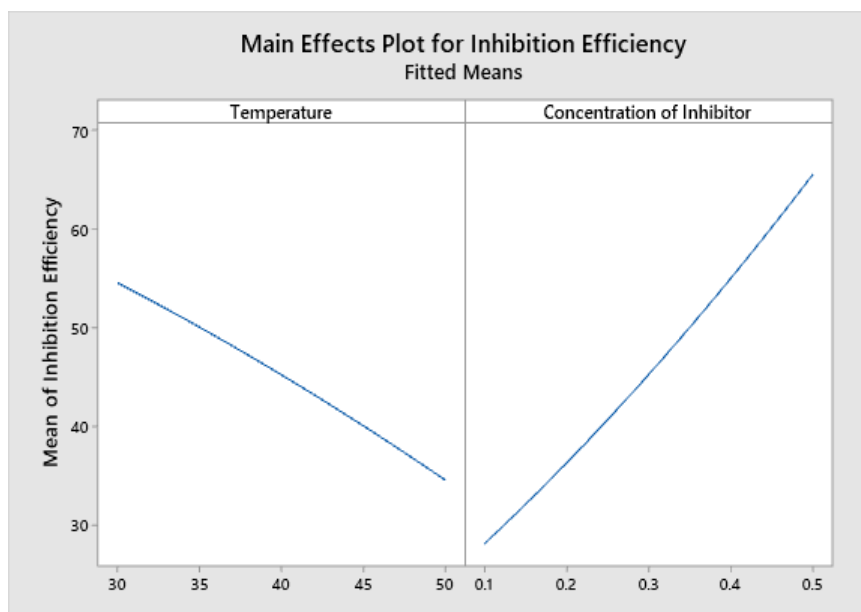


Figure 4: Main Effect Plot for Inhibition Efficiency.

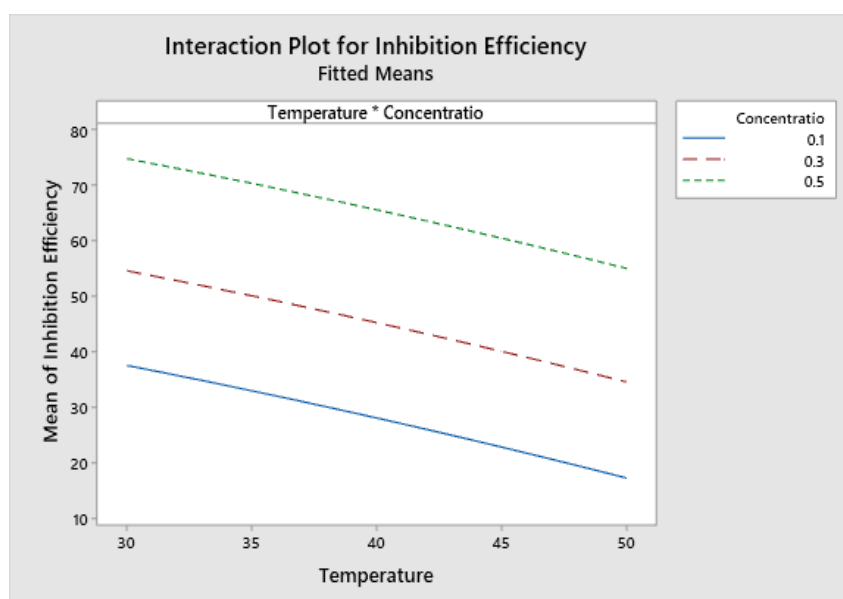


Figure 5: Interaction Plot for Inhibition Efficiency.

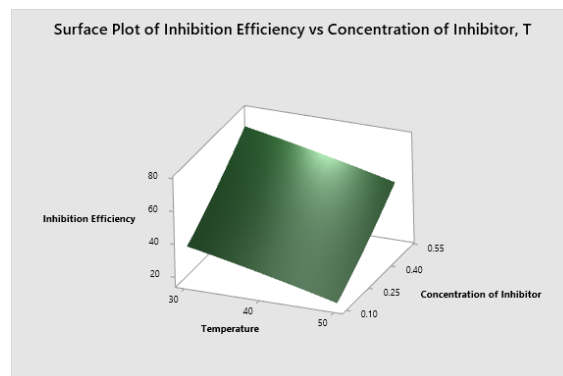
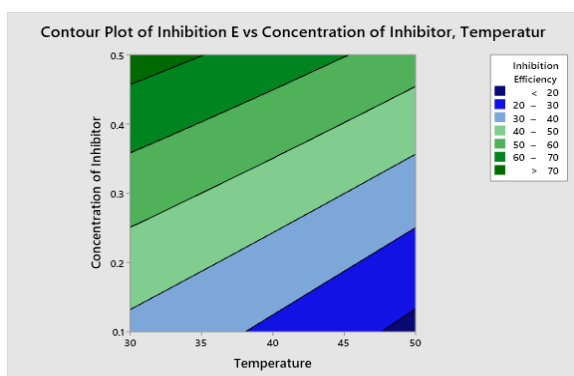


Figure 6: Contour Plot & a Surface Plot for Inhibition Efficiency.

4.2 Response Optimization

Numerical optimization of the model in Equation (4) was done to determine the concentration of inhibitor and temperature, at which, the corrosion rate of 6063 aluminum alloy was at a minimum so that it will results in maximum efficiency. The desirability function approach was used to optimize the process factors for the highest possible inhibition efficiency. The following steps were taken into account before the optimization to detect the measures of numerical optimization. First, the goal factors for temperature and concentration of inhibitor were set while that of inhibition efficiency was set to “maximum”. The predicted optimum parameters of the NaOH environment was estimated to be temperature (30⁰C), and inhibitor concentration (0.5 g/l). At these optimum conditions, the corresponding predicted inhibition efficiency was found to be 74.65%. Confirmation experiments were conducted to validate the optimal parameter settings and to verify the improvement of the inhibition efficiency. The purpose of confirmation experiments is to check the repetitiveness of the experimental results and validate the accuracy of the predictive model.

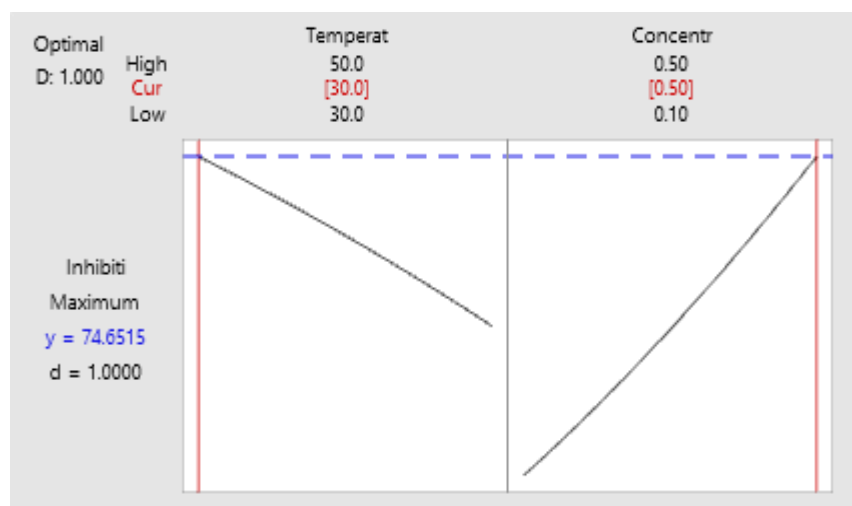


Figure 7: Response Optimization Plot for Inhibition Efficiency.

5. CONCLUSIONS

In this investigation, RSM was utilized to model the inhibition efficiency of 6063 aluminum alloy as an element of the working parameters: temperature and concentration of inhibitor in 0.5M NaOH condition. The subsequent RSM, acquired as a function of noteworthy impacts, was measurably demonstrated to be reasonable. The consequence of the analysis of variance showed that the model was profoundly significant and concentration of inhibitor was the most significant factor with a contribution of 77.21%, affecting the corrosion of 6063 aluminium alloy, thereby increasing the inhibition efficiency. High values of R^2 (0.9921) and adjusted- R^2 (0.99) indicated a good correlation between the predicted and investigational values. The two-factor collaborations were uncovered with 3D response surface plots. In boosting the inhibition efficiency, the ideal working states of the 0.5M NaOH situations were acquired at a temperature of 30⁰C and inhibitor concentration of 0.5 g/l. In this way, it is evident that RSM not just gives a critical understanding of the interaction between the variables, yet also helps to identify the optimum parameter setting for the same. It is inferred that the response surface methodology approach is a promising factual method that could be utilized certainly in foreseeing the ideal working states of 0.5 M NaOH environment, that would maximize the inhibition efficiency of 6063 aluminum alloy used in the chemical industries.

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AUTHORS PROFILE



Dr. Raghavendra Prabhu P is an Associate Professor at the Department of Mechanical & Manufacturing Engineering at Manipal Institute of Technology (MIT), Manipal. As a mechanical engineer, he is actively involved in teaching UG & PG students of MIT and in his capacity as an Assistant Director, Academics he is also responsible for smooth conduct of all the academic related activities at MIT.

Dr. Raghavendra obtained his B.E. (Bachelor of Engineering) in Mechanical Engineering from Manipal Institute of Technology, Mangalore University in 2001, completed M.Tech. (Master of Technology) in Engineering Management from Manipal Academy of Higher Education in 2003. He began his career as a Production Engineer at Manipal Technologies Limited, Manipal in 2003 and later joined as a faculty member in the department of Mechanical & Manufacturing Engineering, MIT Manipal in the year 2005. He obtained PhD from Manipal University, Manipal in the area of Manufacturing Engineering in 2014 under the guidance of Dr. S M Kulkarni, Professor, Dept. of Mechanical Engineering at NITK Surathkal and Dr. S S Sharma, Professor, Dept. of Mechanical Engineering, MIT Manipal. As a faculty member at MIT, Manipal his teaching and research efforts focused in the area of Manufacturing, Process

Optimization and Issues Related to Design and Manufacturing. He was also responsible for setting up of Advanced Material Testing lab at MIT.

Since 2005, he has served the organization for 12 years in various capacities as a faculty member, faculty advisor for various student clubs, sports advisor, in-charge of Advanced Material Testing Lab etc. Dr. Raghavendra is also worked as a Project Head-Functional for the successful implementation of Student Life Cycle Management System at MIT. Dr. Raghavendra has been recognized for his teaching and research with several awards including best teacher award in mechanical engineering stream and best paper awards at various forums. He is a meticulous administrator and also a good sportsman. He has published more than 50 research publications in various national and international journals and conference proceedings.



Dr. Deepa Prabhu is an Assistant Professor Senior Scale at the Department of Chemistry at International Centre for Applied Sciences (ICAS), Manipal. She obtained her Bachelor of Science (Biotechnology) from St. Aloysius College, Mangalore University in 2009, completed M.Sc. Organic Chemistry from Dept. of Science, Manipal Academy of Higher Education in 2011. She began her career as a faculty at Poorna Prajna Pre-University College, Udupi in 2011 and later joined as a Research Scholar under MAHE Structural PhD program from 2011 to 2014. She has Received Best Paper Award in MR-12 in IIT Bombay and Certificate of Appreciation for presentation held at MAHE in 2014. She has Joined as Assistant professor in International Centre for Applied Sciences, MAHE and working since January 2015.

She is awarded with Doctor of Philosophy (Ph.D.) from Manipal Academy of Higher Education in recognition of her research work entitled “Corrosion control of aluminum and 6063 aluminum alloy in phosphoric acid and sodium hydroxide medium with some plant extracts”. She completed her PhD under the guidance of Dr. Padmalatha Rao, Professor, Dept. of Chemistry at MIT Manipal. She has published more than 30 research publications in various national and international journals and conference proceedings. Since 2015, she has served the organization in various capacities as a faculty member, faculty advisor for students and also organizing member for the institutional and University programs.



Dr. Padmalatha Rao is a Professor at the Department of Chemistry at Manipal Institute of Technology, Manipal. She is currently the Coordinator for the Department of Sciences, MAHE. As a faculty member of Chemistry, she has taught PUC and B.Sc. for 3 years, B.Tech. for 31 Years and M.Sc. for 10 years (Electrochemistry, Chemical Kinetics, and Quantum Chemistry). Total of 34 years of teaching experience.

Dr. Padmalatha obtained her B.Sc. from MGM College; Mangalore University in 1984 (VII Rank & Dr. TMA Pai Gold Medal for Science), completed M.Sc. from Mangalore University (II Rank) in 1986. She has received a Young Scientist award from the Indian Council of Chemists for the best research paper in the physical chemistry section in the year 1995 at the Institute of Science Bombay. She obtained her Ph.D. from Mangalore University in 1997 for the thesis “Electrochemical investigation with electrogenerated Ti(III): Analytical, Kinetic and Polymerization studies” under QIP program under the Guidance of Prof. B.S. Sherigara (Former Vice-chancellor Kuvempu University) and Dr. H. V. K. Udupa, (Former Director, Central Electrochemical Research Institute, Karikude, Tamil Nadu).

Her area of research includes electrochemistry and material Science and has guided 4 Ph.D. students and presently 2 students are working under her guidance. The number of M.Sc. projects guided: 04; Ongoing: 02. The total number of publications in national and international journals is 61 out of which 06 are accepted for publication. She has more than 65 conference presented papers under her supervision.